

AF-70



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of:

*Kenneth A. Windhorst*

Examiner: Karl J. Puttlitz

U.S. Serial No. 10/635,983

Group Art Unit: 1621

Filed August 7, 2003

Docket No. C-7220 (CEL-06-7)

For: PROCESSES FOR PREPARING  
ORGANIC COMPOUNDS HAVING  
IMPROVED COLOR CHARACTERISTICS :

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REPLY BRIEF

Sir:

This *Reply Brief* is in response to the *Examiner's Answer*.

REPLY

In the *Examiner's Answer* mailed September 24, 2007, the Examiner has restated the grounds for final rejection which have been treated in Applicant's Main *Brief on Appeal* filed on May 18, 2007. The *Reply Brief* also contains a section entitled "Response to Argument" beginning on page 5. This *Reply Brief* is in response to that section. The Examiner maintains the rejections in this case, based on United States Patent No. 3,214,347 to *Grekel et al.* and now focuses on Example 1 of that reference. Example 1 does not disclose, teach or suggest adding water to an organic composition in order to stabilize color, nor does any other part of that reference; *Grekel et al.*, in fact, teach to **remove water from a product**. The Examiner has misapprehended the reference and misapplied it. The claimed subject matter in this application is **a process** for stabilizing a composition, as pointed out repeatedly by Applicant. Claim 1, for example, reads as follows:

1. (Previously presented) A process for preparation of a color stable organic compound/water mixture from an organic compound selected from the group consisting of C<sub>1</sub> to C<sub>6</sub> carboxylic acids, ketones having boiling points from 154°C to 170°C, and esters having boiling points from about 168°C to about 250°C, the process comprising combining the organic compound with water under conditions of agitation to form a mixed solution of the organic compound and water having a consistent concentration of water comprising from about 100 ppm to about 50,000 ppm water to produce the color stable organic compound/water mixture, wherein the color stable organic compound/water mixture has an APHA color value of 15 or less after being boiled for at least one hour at one atmosphere of pressure.

The claimed subject matter requires 1) a consistent, that is, uniform and unchanging concentration from water and 2) that water is added in the process to form a mixed solution such that the product mixture contains 100 ppm to 50,000 ppm water and 3) that the mixture is color stable, having a color value of 15 or less after boiling for one hour. See Applicant's Main *Brief on Appeal*, page 7 and following. Example 1 of the '347 patent reads in its entirety as follows:

An aqueous mixture of crude acids\* containing .4 percent acetic acid, 5.2 percent propionic acid, 9.2 percent isobutyric acid, 36.8 percent n-butyric acid, 46.5 percent impure C<sub>5</sub> and higher acids, and 1.9 percent of non-acid organic impurities boiling in the range of the iso and n-butyric

acids is subjected to distillation in a conventional fractionation column. The temperature employed at the bottom of the column is about 175° C. (440 mm.) and the top tower temperature is about 132°C. (300 mm.). The crude acid mixture is fed to the column at the rate of 2625 pounds per hour, together with 300 pounds per hour of recycled bottoms from the product n-butyric acid column as described below. Under these conditions, and a reflux ratio of about 10 to 1, a distillate is withdrawn at a rate of 1685 pounds per hour. From the base of the column C<sub>5</sub> and higher acids, together with non-acid impurities, are removed at the rate of 1240 pounds per hour. This stream contains approximately 2 percent of the n-butyric acid present in the original feed. A second column containing only water is then started up under total reflux, after which the aforesaid distillate is used as feed. Water is removed from the column under refluxing conditions until the temperature at a point in the solvent removal zone in the lower part of the fractioning tower reaches a value of about 130°C. (corrected to atmospheric pressure). The temperature at the base of the column is about 164°C. (940 mm.) and the top tower temperature is 100°C. (760 mm.). Distillate is brought overhead, cooled to about 52°C. and allowed to stratify into an upper organic layer consisting essentially of impurities, together with dissolved acids, and a lower aqueous layer containing water-soluble acids. The organic layer is removed from the system at the rate of 145 pounds per hour and contains 4.0 percent of the n-butyric acid present in the aforesaid original feed. The lower aqueous layer is refluxed back to the distillation column. At a point below the feed and above the solvent removal zone, water is introduced at a rate of 25 pounds per hour in order to compensate for the quantity of water removed from the system via the aforesaid organic layer. From the bottom of the tower a substantially dry stream (0.5 weight percent water) of water-soluble C<sub>2</sub>-C<sub>4</sub> acids free from non-acid impurities is withdrawn at the rate of 1565 pounds per hour.

To obtain highly purified n-butyric acid from the dry acid mixture mentioned immediately above, said mixture is fed to a third column operated at a bottom temperature of 157.8°C. (635 mm.) and a top tower temperature of 138.9°C. (500 mm.). A reflux ratio ranging from about 15 to 1 to 23 to 1 is employed. Overhead a stream is withdrawn at the rate of 375 pounds per hour and contains 2.4 percent of the n-butyric acid present in the aforesaid original feed. From the bottom of the column a stream is withdrawn at the rate of 1145 pounds per hour and consists essentially of n-butyric acid. This stream is subjected to further fractionation at a bottoms temperature of 172.2°C. (910 mm.) and at a top tower temperature of 163.3°C. (760 mm.). The reflux ratio in operating this column ranges from 10 to 1 to 20 to 1. Overhead product n-butyric is obtained in 99.2 percent purity, boiling at 162° - 164.2°C. and having a color (APHA scale) of 5. The n-butyric acid thus obtained is removed as distillate at the rate of 890 pounds per hour, while the bottoms fraction from this column is withdrawn at the rate of 300 pounds per hour and

recycled to the original aqueous stream of crude acid feed. Based on the quantity of n-butyric acid present in the original feed, 93.8 percent of the n-butyric acid is recovered by this process.

The following example is included for the purposes of distinguishing the process of our invention over ordinary azeotropic distillation, even with an excess amount of azeotroping agent or entrainer being employed in the latter case.

\*Percent by weight on dry basis.

The Examiner specifically argues that "product disclosed" in Example 1, line 65, is relevant to the claimed subject matter; *Examiner's Answer*, page 6, line 6. A product is not claimed in this case and the Examiner's argument is without any merit, whatsoever. A proposed comparison with a composition in the prior art is irrelevant to this appeal and to the patentability of the claimed subject matter. Using water as reflux it is not a teaching to add water to provide color stability. Indeed, the '347 patent teaches away from adding water, as has been noted in Applicant's Main *Brief on Appeal* at page 8 and following. The entire disclosure of the patent teaches that water is to be removed from the product:

*Grekel et al.* '347 also report in Examples A and B that water is **removed** from the feed. See Table at col. 9, reproduced below:

TABLE

Operating Conditions—Rates— Grams per Hour	Run A			Run B		
Feed.....	323.2			352.9		
Distillate Product—Withdrawn:						
Organic Layer.....	26.7			21.6		
Water Layer.....	0			46.4		
Bottoms.....	308.8			306.3		
Reflux (including internal reflux).....	222.5			197.4		
	Butyric Acid	Non- acids	Water	Butyric Acid	Non- acids	Water
COMPOSITION, WT. PERCENT						
Feed.....	95.0	4.5	0.5	84.3	4.0	11.7
Distillate Organic Layer.....	41.4	53.1	5.5	48.5	44.8	6.7
Distillate Water Layer.....				9.5	2.4	88.1
Reflux.....	All of water layer			9.5	2.4	88.1
Bottoms.....	99.2	0.5	0.25	97.4	2.5	0.11
Plate 30 liquid.....	58.9	2.1	139.0			1.3
COLUMN CONDITIONS						
Barometric Pressure (top column), mm. Hg.....	741.4			754.6		
Temperature, ° C.:						
Plate 55 vapor.....	99.0			99.5		
Plate 40 (feed point vapor).....	99.0			99.5		
Plate 30 vapor <sup>2</sup> .....	98.5			158.0		
Pot vapor.....	158.0			164.0		
Pot liquid.....	163.5			166.5		
MATERIAL BALANCES						
Acid as butyric.....	104			98		
Nonacids by difference.....	109			126		
Water.....	139			103		
Distribution of pure n-butyric acid, percent in bottoms.....	97			95		

<sup>1</sup> In Run A corresponds to 78.0 mol percent and in Run B corresponds to 6.0 mol percent.

<sup>2</sup> Tenth plate below the feed plate.

It is seen in the foregoing Table that in Run A, the feed has 0.5% water, while the bottoms (product) has 0.25% water. In Run B, the feed has 11.7% water and the bottoms (product) has 0.11% water. *Water has not been added—it has been removed.*

The Examiner has apparently ignored the relevant aspects of the reference which teach away from the claimed subject matter and proposes instead a modification of the reference contrary to its express teachings. Furthermore, the Examiner has failed to consider the overwhelming evidence of surprising results submitted in this case.

These are all reversible errors.

Applicant notes the guidelines published in the Federal Register on October 10, 2007 that those parts of the reference which teach away must be considered and that evidence of

unexpected results must likewise be considered. Note the guidelines at page 57529 of Vol.72, No. 195:

Note that combining known prior art elements is not sufficient to render the claimed invention obvious if the results would not have been predictable to one of skill in the art.<sup>48</sup> "When the prior art teaches away from combining certain known elements, discovery of successful means of combining them is more likely to be nonobvious."

<sup>48</sup> *United States v. Adams*, 383 U.S. 39,51-52, 148 USPQ 479, 483 (1966). In *Adams*, the claimed invention was to a battery with one magnesium electrode and one cuprous chloride electrode that could be stored dry and activated by the addition of plain water or salt water. Although magnesium and cuprous chloride were individually known battery components, the Court concluded that the claimed battery was nonobvious. The Court stated that "[d]espite the fact that each of the elements of the Adams battery was well known in the prior art, to combine them as did Adams required that a person reasonably skilled in the prior art must ignore" the teaching away of the prior art that such batteries were impractical and that water-activated batteries were successful only when combined with electrolytes detrimental to the use of magnesium electrodes. *Id.* At 42-43, 50-52, 148 USPQ at 480, 483.

In this case, it is clear that the *Grekel et al.* reference teaches away from the claimed subject matter. Furthermore, the evidence of unexpected superior results warrants allowance.

It is an unexpected and very useful superior result that commercial products' color can be stabilized and enhanced simply by adding relatively low levels of water and maintaining them in the stored product. The results are dramatic. See Examples 5-7, line 17+ of the application filed and paragraphs 4 of the August *Declaration* (reproduced below):

4. The Subject Invention resides, in part, in the discovery that adding water to a finished organic compound can greatly enhance APHA color characteristics. This discovery was unexpected and is very useful since superior color is achieved without expensive additional processing steps. Examples 5-7 of the above-noted application as filed are illustrative:

#### Examples 5-7

The effect of color improvement through the addition of water was determined on three samples from a commercially produced butyric acid run. The samples were prepared by successive distillations of the same



portion of the commercially produced butyric acid run. The APHA colors of the samples were determined to be as follows:

Example 5     13

Example 6     3

Example 7     1

The color variance of the samples is attributable to the fact that more color bodies were present in the first distillation sample as compared to the second and third distillation samples.

To each of these samples was added 20,000 ppm, water while stirring at room temperature, to ensure uniform distribution of the water. Following addition of the water, the APHA colors of the samples were determined as follows:

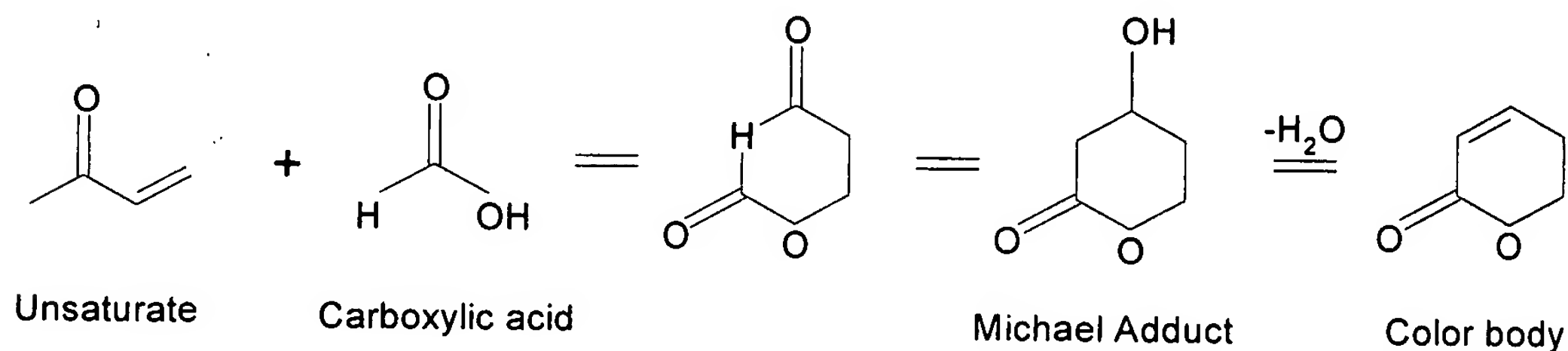
Example 5     1

Example 6     1

Example 7     1

Without intending to be bound by theory, it is believed that the addition of water prevents the formation of color bodies by dehydration. *Note* page 9 of the application as filed, line 3 and following paragraph 5 from the August Declaration:

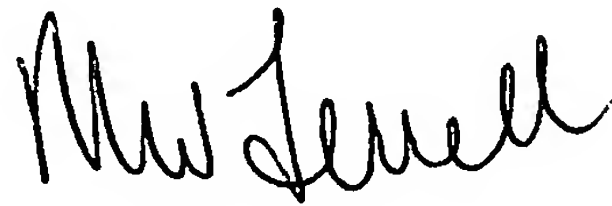
5. It is believed that a compound formed from an unsaturated ketone and a carboxylic acid in the production of the relevant organic compounds leads to formation of a Michael Adduct in accordance with the following reaction process:



As seen from this reaction process, the Michael adduct, upon dehydration, yields color bodies thought to lead to the undesirable darker color products. It is believed that by adding water to the organic compound products, formation of the color bodies is prevented.

For the above stated reasons and for the reasons noted in Applicant's Main *Brief on Appeal* filed May 18, 2007, all outstanding rejections in this case should be reversed and this case passed to issue.

Respectfully submitted,

A handwritten signature in black ink, appearing to read "Michael W. Ferrell". The signature is fluid and cursive, with the first name "Michael" and last name "Ferrell" being clearly distinguishable.

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November 5, 2007



**CERTIFICATE OF MAILING BY FIRST CLASS MAIL (37 CFR 1.8)**Applicant(s): **Kenneth A. Windhorst**

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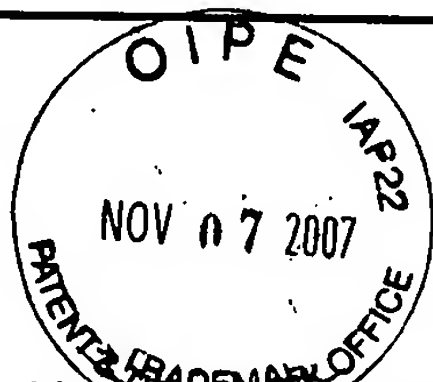
Examiner

**Karl J. Puttlitz**

Customer No.

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Group Art Unit

**1621**Invention: **PROCESSES FOR PREPARING ORGANIC COMPOUNDS HAVING IMPROVED COLOR CHARACTERISTICS**I hereby certify that this **Reply Brief and return postcard***(Identify type of correspondence)*

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*(Date)***Carol R. Maddaloni***(Typed or Printed Name of Person Mailing Correspondence)*

A handwritten signature in cursive script, appearing to read "Carol R. Maddaloni".

*(Signature of Person Mailing Correspondence)***Note: Each paper must have its own certificate of mailing.**